https://dx.doi.org/10.24001/ijaems.3.6.14 *ISSN: 2454-1311*

Experimental study and calculation of the electron transfer coefficients on the dissolution behavior of chitosan in organic acids

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Abstract*—Chitosan (CH) consists of water-insoluble N-acetylglucosamine and D-glucosamine molecules and has a higher solubility at a pH below six. This studyevaluated the solubility of chitosan in solutions of organic acids for the formation of films.* HyperChem™*software was used to perform the quantum analysis. In the experimental trials, the total soluble mass (TSM) and the viscosity of the solutions were measured by capillary viscometer. The chitosan filmswere made by the plate melting method, and the filmcharacteristics were evaluated. A quantum simulation suggested that lactic acid (LA) has a greater stability to react with chitosan. It was then verified experimentally that LA is a better solvent for chitosan due to the increase in its viscosity. The chemical interaction between CH and LA in solution favors the polymerization of films with better physical properties. We thereforeconclude that the uniformity in the formation of films of this polymer depends on the chemical interaction between the CH and the acid and not on the degree of solubility of the polymer.*

Keywords*—*Chitosan, Electron Transfer Coefficient (ETC), HyperChem, Organic Acid, Quantum Chemistry

I. INTRODUCTION

CH is a natural cationic polymer formed of molecules of N-acetyl glucosamine and D-glucosamine, derived from chitin [1]. It has insoluble D-glucosamine units at neutral and alkaline pH.

CH is soluble in aqueous solutions of pH less than 6.5 [2-3]. At a low pH, the amino groups in the polymer chain of CH are protonated, resulting in a water-soluble cationic polyelectrolyte [4]. It can dissolve in organic and inorganic acids [5-6]. The solution of CHin aqueous solutions of acids depends on the concentration of protons and must be proportional to the concentration of the amino groups in solution [7].

In prior studies, the researchers used CHas a semipermeable matrix in biological dressings and films for the treatment of wounds and burns. The properties of CHare antimicrobial, hemostatic, anti-inflammatory, non-toxic, biodegradable and biocompatible [8-9]. Finding easily manufactured, stable, resistant, compliant CH-based films can have important additional applications.

The effect of viscosity on the solubility of Chitosan.

In solution, hydrogen bonds between CH molecules and water determine the viscosity of the solution. The acids have a hydrophilic structure that form strong hydrogen bonds. Strong hydrogen bonds and CH decrease molecular mobility and increase the viscosity of the solution [10].

The solubility of Chitosan in Organic Acids.

Viscosity measures the ability of a polymer to enhance the viscosity of a solvent at a given temperature. Capillary viscometers are useful for evaluating the viscosity of a polymer. The technique of viscometry consists of recording the time required by two fluids to flow between two reference marks in a capillary tube at a constant temperature [11].

The objective of this work was to evaluate the solubility of CH in solutions of organic acids by quantum simulation and experimental validation for the formation of chitosan films with optimal physical characteristics.

II. MATERIALS AND METHOD

Quantum analysis

We quantitatively analyzed the solubility of CH in aqueous solutions of organic acids for the formation of films using the program Hyper Chemmolecular simulator for Windows (Serial # 12-800-1501800080, Multi ONI nsurgentes Sur 1236 Tlacoquemecatl Col. del Valle, Benito Juárez, DF, CP 03200. Mexico). HyperChem™ is

https://dx.doi.org/10.24001/ijaems.3.6.14 *ISSN: 2454-1311*

a quantum modeling program. The function of Hyperchem is to analyze the compounds chemically and their possible interactions [12-13].

Hyper Chem™.The selected method of analysis was the Semiempirical Method Parametric 3 (SE-PM3). The calculation was optimized with thePolakRibiere method, calculating the following variables: HOMO-LUMO, Bandgap (BG), Electrostatic Potential (EP) and other properties[12-13].

Selected specific parameters for each of the simulations shows in Table 1 and 2.

Table.1: Parameters used for quantum computing molecular orbitals HOMO and LUMO and BG

Parameter	Value	Parameter	Value
Total Charge	0	Polarizability	Not
Spin Multiplicity	1	GeometryOptimization: Algoritm	Polak-Ribiere (Conjugated gradient)
Spin Pairing	RHF	Termination condition RMS gradient of	0.1 kcal/Amol
StateLowestConvergetLimit	0.01	Terminationconditionor	195 maximumcycles
InterationLimit	50	Terminationconditionor	In vacuo
AccelerateConvergence	Yes	Screenrefreshperiod	1 cyclest

Table.2: Parameters used for quantum computing E-, E+ and EP

Parameter	Value	Parameter	Value
Molecular Property	PropertyElectrostaticPotential	ContourGridIncrement	0.05
Representation	3D MappedIsosurface	MappedFunctionOptions	Default
Isosurface Grid: Grid Mesh Size	Coarse	Transparencylevel	A criteria
IsosurfaceGrid: GridLayaout	Default	Isosurface Rendering: Total charge density contour value	0.015
ContourGrid: StartingValue	Default	Rendering Wire Mesh	Default

[12]

Experimental analysis

The solubility of CH in aqueous solutions of organic acids for the formation of filmswas evaluated as follows:

1% low molecular weight CH (Sigma-Aldrich) was added in 20mL of 0.1 M solution of organic acids in duplicate (Table 3). The solution was dissolve at 25°C, with magnetic stirring at 660 rpm for 15 minutes, and the final solution was filtered [14-15].

The CH films were made by the plate casting method [16]. The filtrate was poured into a polypropylene vessel and placed in a drying oven at 80 °C for three hours.

To determine the solubility of CH in the organic acids, the total soluble mass (TSM) of the solutions was measured[17] The castings were dried at 80°C until steady-stateweight was recorded of the filter paper with the undissolved residue of CH. The percentage of TSM was calculated using the following formula (*Equation 1*):

$$\%TSM = \frac{Q_{i(g)} - Q_{nd(g)}}{Q_{i(g)}} \qquad \textit{Equation 1}$$

Where:

Q_i *= initial weight of CH*

Q_{nd} *= weight of the undissolved portion of the CH*

Table.3: Organic Acids used in the solubility tests of CH

Key	Acids (0.1 M)
AcA	Acetic Acid
AsA	Ascorbic Acid
CA	Citric Acid
MA	Malic Acid
LA	Lactic Acid

The solutions of CH with the acids that presented the best solubility were AcA and LA. The identified solutionswere used to to make CH films. The drying was carried out on a plate covered with low-density polyethylene. Film thickness was measured (Micrometer MITUTOYO), as well as planar dimensions, weight (OHAUS Sensitivity: 0.01 g) and density of the films [18].

We measured capillary viscosityin the samples that presented better solubility (AcA + CH and LA + CH)[19]. 9 ml of solutions AcA + CH, LA + CH, and tri-distilled water(positive control) were placed in 15 ml falcon tubes. The tubes are placed in a universal support vertically. Capillary tubes in the center of each falcon tube, and the upper limit of a perforated rubber stopper (No. 6, Marca Nacional).The setup was left at room temperature (25 °C) for one hour. The height of capillary plus stopperwas7.5

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cm. Finally, the height of the liquid in each capillary was recorded in triplicate under these conditions.

III. RESULTS AND DISCUSSION

Quantum chemical results.

Table 4 shows a study of reactivity and chemical stability between two compounds from the perspective of quantum analysis.

We calculated the ETC[12]of each substance by dividing the BG between the EP. The calculated ETC represents the number of multiples an electron's EP is needed to jump the BG between molecules of the same compound. The compound that has a higher ETC has greater impediment for the intermolecular movement of its electrons. Therefore, CH is the most stable and least reactive compound due to its lower ETC, while water is the most reactive and least stable of these molecules.

Table.4: Chemical Stability of Compounds

COMPOUND	HOMO	LUMO	BG	E-	E+	EP	ETC
CH	-9.503	1.786	11.290	-0.106	0.200	0.306	36.894
CA	-11.271	0.208	11.479	-0.103	0.203	0.306	37.514
AcA	-11.256	1.025	12.281	-0.128	0.198	0.326	37.672
MA	-11.453	0.430	11.883	-0.119	0.190	0.309	38.456
AsA	-10.626	0.662	11.288	-0.112	0.172	0.284	39.747
LA	-11.350	0.970	12.320	-0.131	0.178	0.309	39.872
W	-12.316	4.059	16.375	-0.129	0.173	0.302	54.221

Table 5 shows the results of all possible cross-bands of all compounds studied. This crossing of bands is done by taking the HOMO of a substance with the LUMO of the other substance and calculating its BG. In the same way, the EP of each substance is calculated.

Compounds with their lowest ETC have the highest probability of interacting by dissolving or reacting. In this case, we foundthat the organic acids most reactive with CH are CA and MA. LA has a more stable reactivity (Figure 1).

As shown in the same Table 5, all the acids have a high probability of electron exchange with CH.

In Figure 1, the dots represent the bottom of the quantum well of each interaction; while the dotted lines represent the bottoms of the quantum wells upper limit (W) and lower limit (CH). It can be deduced that acids in aqueous solution easily attack CH by removing electrons, *i.e.* as oxidizing agents. From Table 5 and Figure 1 we can suggest that CA attacks CH more easily. This type of attack can range from Van der Waals forces, H-bridges, to chemical reactions in the electron orbitals.

Table.5: Chemical Reactivity of Compounds. Organic acids against CH

Reducing Agent	Oxidizing Agent	HOMO	LUMO	BG	E-	E+	EP	ETC
CH	CA	-9.503	0.208	9.711	-0.106	0.203	0.309	31.429
CH	MA	-9.503	0.430	9.933	-0.106	0.190	0.296	33.557
CH	AcA	-9.503	1.025	10.528	-0.106	0.198	0.304	34.631
CH	AsA	-9.503	0.662	10.165	-0.106	0.172	0.278	36.566
CH	LA	-9.503	0.970	10.473	-0.106	0.170	0.280	36.878
CH	CH	-9.503	1.787	11.290	-0.106	0.200	0.306	36.895
CA	CA	-11.271	0.208	11.479	-0.103	0.203	0.306	37.514
AcA	AcA	-11.256	1.025	12.281	-0.128	0.198	0.326	37.672
W	CA	-12.316	0.208	12.524	-0.129	0.203	0.332	37.724
MA	MA	-11.453	0.430	11.883	-0.119	0.190	0.309	38.456
LA	CH	-	1.7	13.	-	0.2	0.3	39.

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		11.350	87	137	0.131	00	31	689
AsA	AsA	-10.626	0.662	11.288	-0.112	0.172	0.284	39.748
AcA	CH	-11.256	1.787	13.043	-0.128	0.200	0.328	39.765
AsA	CH	-10.626	1.787	12.413	-0.112	0.200	0.312	39.784
LA	LA	-11.350	0.970	12.321	-0.131	0.178	0.309	39.872
W	MA	-12.316	0.430	12.746	-0.129	0.190	0.319	39.956
W	AcA	-12.316	1.025	13.341	-0.129	0.198	0.327	40.798
MA	CH	-11.453	1.787	13.240	-0.119	0.200	0.319	41.505
W	CH	-12.316	1.787	14.103	-0.129	0.200	0.329	42.866
CA	CH	-11.271	1.787	13.058	-0.103	0.200	0.303	43.095
W	AsA	-12.316	0.662	12.978	-0.129	0.172	0.301	43.118
W	LA	-12.316	0.970	13.286	-0.129	0.178	0.307	43.278
CH	W	-9.503	4.059	13.562	-0.106	0.173	0.279	48.609
LA	W	-11.350	4.059	15.409	-0.131	0.173	0.304	50.687
AcA	W	-11.256	4.059	15.315	-0.128	0.173	0.301	50.880
AsA	W	-10.626	4.059	14.685	-0.112	0.173	0.285	51.525
MA	W	-11.453	4.059	15.512	-0.119	0.173	0.292	53.123
W	W	-12.316	4.059	16.375	-0.129	0.173	0.302	54.221
		316			29			
CA	W	-11.271	4.059	15.330	-0.103	0.173	0.276	55.542

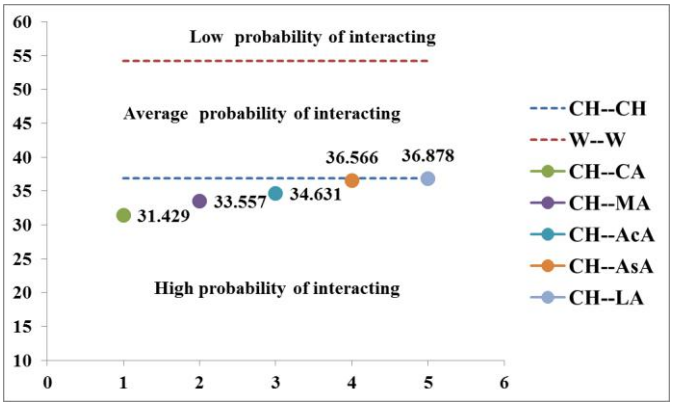

Fig.1: Background of quantum wells represented by points. It can be noted that the five acids that have a high probability of electron exchange.

LA was identifiedas one of the organic acids with the greatest potential for interaction within quantum stability analysis (Figure 2)[20-21]. The simulations and subsequent experiments show chemical interaction of CH with LA.

Experimental results.

To validate the computational simulations, physical experiments were performed with the following results: The solutions with the highest percentage of TSM of CH were AcA (ETC = 34.631), LA (ETC = 36.878) and MA (ETC = 33.557) with 89, 79 and 72% respectively. The solutions with a lower proportion ofTSM were CA (ETC = 31.429) and AsA (ETC = 36.566), with 55 and 65% respectively (Table 6).

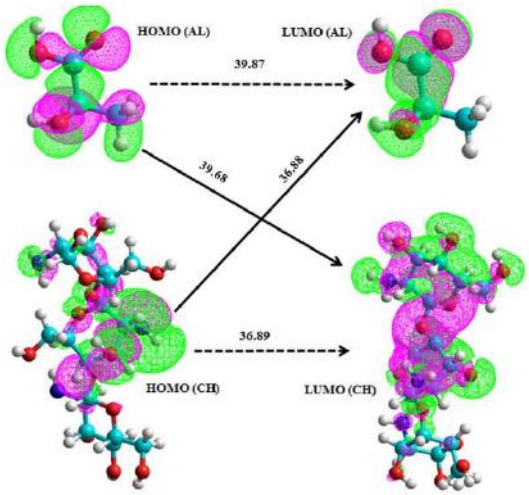

Fig.2: Interaction of CH vs. LA. In this figure it can be seen that the HOMO (CH) vs. LUMO (AL) competes with the normal band of the CH. Therefore,it is interpreted as AL dissolves CH.

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CH was soluble in the MA solution (TSM: 72%). However, it is possible that this acid changes the chemical properties of CH, and therefore does not polymerize (Table 7).

In the quantum analysis, a high reactivity of MA and AsAwith CH (Table 4/5)was observed. However, the solutions of these acids formed films with non-favorable physical characteristics. Therefore, they have an adverse effect on the polymerization of CH (Table 7).There is a possiblechemical reaction CH--MA and CH—AsA (Figure 1)

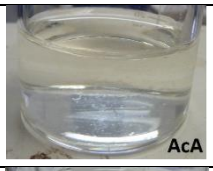
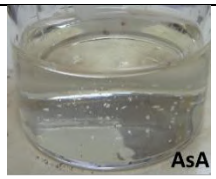
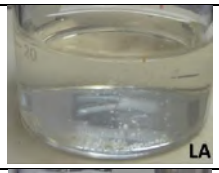
CH in the CA solution hadlow solubility (TSM: 55%) but formed a uniform film (Table 7). The characteristic may derive fromthe high chemical interaction between CAadCH as observed in the quantum analysis (Table 4/5). The AsA solution hadhigh solubility (TSM: 65%). However, the film was hard, nonresistant and presented oxidation. AsA is a thermolabile vitamin, so it is possible that it reacted with the drying temperature (Table 7).

The CH films of better flexibility, appearance, thickness and resistance were those that we formed from the LA, AcA and CA. These acids had 79, 89 and 55% of TSM, respectively. As a result, the uniformity in the formation of CH films didnot depend on the amount of CH dissolved but the chemical interaction between CH and acid in solution (Table 6/7).

The solutions of AcA and LAhada soft amber color. The AcAsolution had a characteristic vinegar odor, and that of LAwas odorless. CHdissolvedin AcA and LA solutions allowed the formation of smooth and resistant films at different temperatures (Table 8).

Solutions of both acids dried at 100 °C formed the densest films. The highest density CH film was with AcA solution (1.67 g / cm3) at 100 °C, and the lowest density was with LA solution (0.66 g / cm3). Therefore, the thickness and density of the films werenot proportional (Table 8).

Table.6: TSMof Chitosan CH in solutions of organic acids

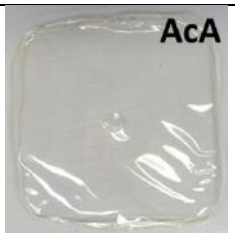
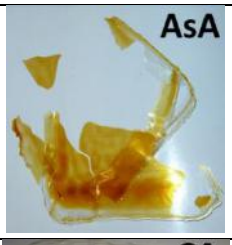
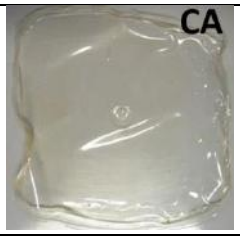
Solution (A 0.1M +CH1%)	Solubility	TSM (%)
	*	89.0
	*	65.0
	*	55.0
	*	79.0
	*	72%

*Soluble

The drying temperature at 100 °C allowed the formation of thinner films in both samples. It was observed that the thickness of the film was dependent on the volume and drying temperature of the solution. This information was versatile in the applications of CH films (Table 8).

The capillary viscometryresults for the solutions of AcA and LAwere statistically analyzed. The displacement reachedby the solution of AcA in the capillary viscometer suggests that it was less viscous than LA. Overall, theviscometrytests suggest that LA was a better solvent for CH by increasing solution viscosity (Table 9) [22-23].

Table.7: Physical Characteristics of CH Films Dissolved in Solutions of Organic Acids

CH Films	Physical characteristics
	Transparent Odor (vinegar) Flexible, Lisa, R, Hard, Thick, Uniform
	Amber (oxidation), Lisa, NR, Hard Thick, Uniform
	Smooth amber yellow, Lisa, R, Hard, Flexible Thick, Uniform

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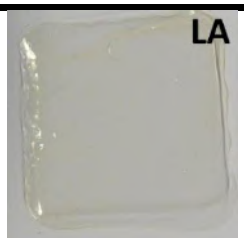	Smooth amber yellow, Lisa, R, Hard, Flexible Thick, Uniform
	* Precipitate solid coffee

* No film formed; R: Resistant; NR: Not resistant

CH wasmore soluble in AcA (TSM: 89%) than LA (TSM: 79%). However, the chemical interaction between CH and LA in solution favors the polymerization of films with better physical properties. In simulation, the AcA had a higher reactive interaction than LA, but in the experimental validation, the LA had better final physical properties.

Table.8: CH films soluble in solutions of AcA and LA after drying at 100 °C (A) and 90 °C (B)

Sample	A:LA	B:LA	A:AcA	B:AcA
				
Dimensions (cm)	5.4x13.0	6.0x13.1	4.2x9.8	4.1x7.3
Weight (g)	0.5943	0.4526	0.1647	0.1890
Physicalcharacteristics	Amber yellow faint, odorless, Soft thin, elastic, uniform	Soft amber, odorless Soft thick, elastic	Transparent, with odor (vinegar), hard, uniform	Transparent, with odor (vinegar), hard, uniform

Table.9: Capillary viscometrytest in AcA and LAsolutions (0.1 M)

Sample	Height reached in capillary (t=1 h)	Viscosity
AcA	2.8 ± 0.17	*
LA	1.8 ± 0	**
W	3.0 ± 0.29	-

- Not viscous * Less viscous ** More viscous; W:Water

IV. CONCLUSION

CH dissolved in a solution of MAand AsAdidnot allow the formation of uniform films. The MA solution was soluble but was likely to degrade the CH.

The acids with the best characteristics for the formation of optimal films werelactic, acetic and citric acid.

The quality of the films of CHdidnot correspond to the percentage of solubility of CH in the acid but corresponded to the chemical interaction between the chemical structure of both.

Quantum simulations suggested that LA has a greater abilityto react with CH. LA was a viable option for interaction with CH.

The chemical interaction during the polymerization process between the solution of 1% CH and 0.1 M LAwas better because of its high viscosity.

LA and AcA solutions dissolve CH for the formation of films with optimal physical characteristics.

CH is more soluble in AcA. However, the physical properties of the film formed from LA are better.

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